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H. W. WILEY, Chief of Bureau.

METHODS FOR THE ANALYSIS OF MAPLE PRODUCTS AND THE DETECTION OF ADULTERANTS, TOGETHER WITH THE INTERPRETATION OF THE RESULTS OBTAINED.

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INTRODUCTION.

This circular contains a compilation of the methods of analysis of maple products prepared for the convenience of food analysts, together with such a discussion of the average results obtained as may serve to guide others in the interpretation of their results and the use of the methods. In the preparation of this circular the following authorities have been consulted freely, in some cases the methods being given exactly as in the original, while in others modifications suggested by work in the Sugar Laboratory have been introduced: The work of C. H. Jones and his assistants as published in the Seventeenth and Eighteenth Annual Reports of the Vermont Agricultural Experiment Station (1904–5); Julius Hortvet's article on "The Chemical Composition of Maple Sirup and Maple Sugar—Methods of Analyses and Detection of Adulteration," published in Journal of the American Chemical Society, 1904, volume 26, page 1523, and a paper by A. L. Winton, entitled "A Method for the Determination of the Lead Number in Maple Sirup and Maple Sugar," published in the same journal in 1906 (volume 28, page 1204). The correlation of this work and its publication in one place it is thought will be of considerable convenience and value especially for those engaged in food inspection work.

Respectfully,

H. W. Wiley,
Chief of Bureau.

Approved:
James Wilson,
Secretary of Agriculture.

Washington, D. C., *July 6, 1908.*

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CHARACTERISTICS OF MAPLE PRODUCTS.

The following quotations from Jones, of the Vermont station, serve to characterize maple products from the point of view of both the consumer and the chemist:

> Maple sugar is a crude product which from the nature of the case it is impracticable to refine. Neither is such refining desirable, for the peculiar value of maple goods is due, not to their sugar content or sweetness, but rather to the agreeable "maple flavor" that accompanies them. Too much refining eliminates this flavor and then the value of the goods is no greater than that of ordinary granulated or brown sugar. * * *
>
> Pure maple products and those adulterated with cane sugar seem most clearly differentiated by the varying amounts and nature of their ash contents, namely, the solubility and alkalinity of the soluble and insoluble ash. Only a trace of ash is found in refined cane sugar, while in maple sugar a much larger quantity is normally present. Commercial brown sugar may also carry a considerable ash content, but its character is so distinct from that found in the maple that mere similarity in amount of ash does not seriously interfere with its detection.

Again, pure maple products give a large voluminous precipitate with lead subacetate, while brown sugars give only a small amount, and white sugars none. Various authors have proposed methods for detecting admixtures of adulterants based upon this fact. Upon these two facts the chemist depends principally for the detection of adulteration. Mr. Jones further says: "The color of maple products is no indication of purity. Pure goods may be almost snow white, or they may be nearly jet black or any intermediate shade of brown. The color of pure goods is largely dependent on the methods used in handling and boiling the sap." In general the first run of sap produces a much lighter colored sirup and sugar than the later runs.

The odor of the product is of little value in determining the purity. Fresh goods have the very characteristic odor, but on standing or in dry samples of sugar the odor is apt to be "bound up."

The taste is likewise of little value, as the flavor of the products from different sections of the country varies in strength. Jones gives the following characteristics of maple products from various localities: Pennsylvania—sweet and flat (often like molasses) maple flavor; Ohio—mild, delicate (almost to flatness) maple flavor; Vermont—mild, delicate, good maple flavor; New York—strong maple flavor; Canada—good maple flavor, dark in color with strong flavor.

DETERMINATIONS TO BE MADE.

From this discussion it is seen that ordinarily it is sufficient to make the following determinations:

1. Moisture.
2. Polarization at 20° C. before and after inversion.
3. Percentage of reducing sugars, figured as invert sugar.
4. Winton's lead number.
5. Percentage of ash (total).
6. Percentage of soluble and insoluble ash.

7. Alkalinity of soluble ash.
8. Alkalinity of insoluble ash.
9. Ratio of insoluble ash to soluble ash.

Other determinations that might be made if the analyst deemed it advisable are:

10. Reaction with iodin after decolorizing with charcoal.
11. Polarization before and after inversion at 87°.
12. Malic acid value.
13. Percentage of calcium oxid (CaO), potassium oxid (K_2O), and sulphur trioxid (SO_3) in the ash.

METHODS OF ANALYSIS.

The methods for making these determinations are given in the order in which they have been enumerated, and in connection with each method the average figures obtained by it for pure maple sugar and sirup, based on the published works of Jones, Hortvet, and Winton and on about sixty samples analyzed in 1904 and 1905 in the Bureau of Chemistry, are stated.

I. PER CENT OF MOISTURE AND DRY SUBSTANCE.

(*a*) Determine loss of weight by drying 4 grams of the sample on sand in a vacuum oven at 70° C. to constant weight.

(*b*) Add 100 grams of water to 100 grams of sirup and obtain the degrees Brix with a Brix hydrometer. Then multiply this by 2 and subtract from 100 to obtain the per cent of water.

(*c*) Find the refractive index of the sirup at 20° with an Abbe heatable prism refractometer and consult tables of Geerlig in the International Sugar Journal, February, 1908, page 69, or in the Journal of the American Chemical Society, September, 1908. The per cent of moisture is obtained by subtracting the per cent of dry substance from 100.

The Food Standards[a] allow not more than 32 per cent of water in maple sirup, which would be equivalent to a specific gravity of 1.3383. No standard water content is set for maple sugar. Hortvet publishes figures for water content of pure maple sugar varying from 4.27 per cent to 15.67 per cent, giving an average of 8.85 per cent.

II. POLARIZATION AT 20° C.

Make up the normal weight (26 grams) of the sample to 100 cc (true) in a flask. If the original substance is too dark to read easily, clarify with lead subacetate. Polarize in a 200 mm tube. Free 50 cc of solution from lead by treating with anhydrous sodium carbonate or neutral potassium oxalate and invert by either method described in Bulletin 107, Revised.[b] Make the reading in a 200 mm tube, correct for dilution, and calculate the percentage of sucrose by the following formula:

$$S = \frac{100\,(P - 1)}{142.66 - \frac{T}{2}}$$

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[a] U. S. Dept. Agr., Office of the Secretary, Cir. No. 19, p. 10.

[b] U. S. Dept. Agr., Bureau of Chemistry, Methods of Analysis, Bul. 107, Revised, p. 40 (c).

Hortvet gives the following figures for pure maple sugars:

Direct polarization (°V.)	+72.6 to +87.4
Invert polarization (°V.)	−31.0 to −27.2
Sucrose by Clerget method (per cent)	80.20 to 89.53

For pure maple sirups:

Direct polarization (°V.)	+51.0 to +62.2
Invert polarization (°V.)	−25.6 to −20.0
Sucrose by Clerget method (per cent)	53.29 to 65.11

III. REDUCING SUGARS.

This determination can be made on a portion of the lead-free solution obtained from Method II, but it would be better in case much reducing sugar is present to make up 5 grams of the sample to 100 cc, clarifying with about 1 cc of neutral lead acetate. After filtering, remove the lead with anhydrous sodium carbonate or potassium oxalate. Determine the reducing sugars in 25 cc by the Munson and Walker method,[a] or by the Allihn method.[b] The reducing sugar should be calculated as invert sugar, as given in Munson and Walker's table; or, if Allihn's method is used, multiply the percentage of dextrose by the factor 1.044 to obtain the per cent of invert sugar. For accurate determinations of reducing sugar in maple sugar, use Meissl and Hiller's table,[c] or with Munson and Walker's method prepare the sugar solution of the strength given in either of the two columns headed "Invert sugar and sucrose."

In nineteen samples of pure maple sugar analyzed by Hortvet, eleven samples were below 3 per cent, three samples between 3 and 4 per cent, and two samples between 4 and 5 per cent. The remaining three samples were above 5 per cent. The highest result obtained was 8.37 per cent.

Among twenty-two samples of pure maple sirup analyzed by Hortvet, ten were below 2 per cent, five between 2 and 3 per cent, three between 3 and 4 per cent, and two between 4 and 5 per cent. The highest result was 9.17 per cent.

IV. LEAD NUMBER (WINTON).[d]

For this determination a standard lead subacetate solution must be prepared according to the following directions:

Standard lead subacetate solution.—Boil for one-half hour 430 grams of normal lead acetate and 130 grams of litharge with 1,000 cc of water; cool the mixture; allow to settle, and dilute the supernatant liquid to 1.25 specific gravity. To a measured amount of this solution add four volumes of water and filter if not perfectly clear.

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[a] J. Amer. Chem. Soc., 1906, *28:* 663–686; U. S. Dept. Agr., Bureau of Chemistry, Bul. 107, Revised, p. 241.

[b] U. S. Dept. Agr., Bureau of Chemistry, Bul. 107, Revised, p. 49–51.

[c] Ibid., p. 44.

[d] J. Amer. Chem. Soc., 1906, *28:* 1204.

The solution should be standardized each time a set of determinations is made, although it is claimed that there is no deterioration in this solution for a month.

Description of method.—Weigh 25 grams of the material (or 26 grams, if it is desired to determine sugars by polariscope in the same portion) into a 100 cc flask. Add 25 cc of standard lead subacetate solution, fill to the mark, shake, allow to stand at least one hour, and filter. From the clear filtrate pipette off 10 cc, dilute to 50 cc, and add a moderate excess of sulphuric acid and 100 cc of 95 per cent alcohol. Let stand over night, filter on a Gooch crucible, wash with 95 per cent alcohol, dry at a moderate heat, ignite at low redness for three minutes, taking care to avoid the reducing cone of the flame, and weigh. Calculate the amount of lead in the lead sulphate precipitate by the factor 0.6829, subtract the result from the amount in 2.5 cc of the standard solution (or from the amount found in the blank), and the figure obtained will be the amount of lead contained in the lead precipitate of organic matter. Divide this number by 2.5 and multiply by 100, to obtain the *lead number* or the per cent of lead precipitated by 1 gram of the original sample. (In case 26 grams are used, this last divisor should be 2.6 instead of 2.5.)

The figures for pure maple sugar vary from 1.83 to 2.48; average, 2.23.

The figures for pure maple sirups vary from 1.19 to 2.03; average, 1.49.

Adulterated products are generally much lower.

V. PER CENT ASH (TOTAL).

Weigh 5 grams of sirup or sugar into a tared platinum dish; heat over asbestos board until the contents are thoroughly carbonized; transfer to a muffle and burn at *low red heat* to a white or gray ash. Cool on desiccator and weigh quickly. The final ashing can be accomplished over an ordinary bunsen burner, and in most cases this is to be preferred to a muffle, as there is less danger of loss of ash by overheating. If considerable frothing occurs during the first drying and burning, place a few drops of pure olive oil on the sample during burning. Divide the weight of ash by 5 and multiply by 100 to obtain the percentage of ash.

The rate of burning and kind of ash are indicative of purity. Adulterated articles, as a rule, take longer to burn and produce an ash hard to rid of the final particles of carbon. Pure maple ash is grayish, sometimes greenish, in color and often has a network structure like the veining of a leaf. According to the food standards, maple sugar should not contain less than 0.65 per cent of ash and maple sirup not less than 0.45 per cent of ash. The average figures compiled from the sources mentioned are as follows:

Pure maple sugar—percentage of total ash:

Jones	0.64 to 1.32; average, 0.91
Hortvet	0.65 to 1.30; average, 0.91

Pure maple sirup—percentage of total ash:

Jones	0.50 to 0.86; average, 0.60
Hortvet	0.52 to 1.01; average, 0.68
Bureau of Chemistry	0.46 to 0.86; average, 0.60

VI. PER CENT OF SOLUBLE AND INSOLUBLE ASH.

To the platinum dish containing the ash (from determination V) add 40 cc of hot water and boil gently for two minutes, using care to avoid spattering. Filter through a small ashless filter and wash with hot water until the filtrate amounts to about 100 cc. Retain the filtrate for making determination VII, alkalinity of soluble ash.

Transfer the filter paper containing the insoluble ash to the same platinum dish and carefully ash at a low red heat, as before. Cool and weigh. The increase in weight over the platinum dish is due to the insoluble ash. Divide this quantity by 5 and multiply by 100 and the percentage obtained is the *insoluble ash.* Subtract this per cent of insoluble ash from the per cent of total ash obtained in determination V and the result is the per cent of soluble ash. To check this figure, the percentage of soluble ash can be obtained direct by evaporating the 100 cc of water to dryness and weighing. Save the platinum dish with the insoluble ash for determination VIII, alkalinity of insoluble ash.

The following average results have been compiled:

Pure maple sugar—percentage of soluble ash:

Jones	0.36 to 0.63;	average, 0.48
Hortvet	0.33 to 0.67;	average, 0.45

Pure maple sirup—percentage of soluble ash:

Jones	0.29 to 0.47;	average, 0.38
Hortvet	0.21 to 0.49;	average, 0.39
Bureau of Chemistry	0.25 to 0.63;	average, 0.38

Pure maple sugar—percentage of insoluble ash:

Jones	0.20 to 0.87;	average, 0.43
Hortvet	0.32 to 0.78;	average, 0.53

Pure maple sirup—percentage of insoluble ash:

Jones	0.16 to 0.51;	average, 0.22
Hortvet	0.15 to 0.55;	average, 0.31
Bureau of Chemistry	0.14 to 0.56;	average, 0.22

VII. ALKALINITY OF SOLUBLE ASH.

Transfer the 100 cc of water solution from the preceding determination to a beaker or porcelain evaporating dish and determine the alkalinity by titrating with tenth-normal hydrochloric acid, using methyl orange as an indicator. The number of cubic centimeters of acid used divided by 5 gives the number of cubic centimeters of tenth-normal acid necessary to neutralize the ash of 1 gram of sample, which figure is used to express the alkalinity of the soluble ash.

Pure maple sugar—cc of acid necessary to neutralize 1 gram:

Jones	0.40 to 0.80;	average, 0.61
Hortvet	0.55 to 0.95;	average, 0.66

Pure maple sirup—cc of acid necessary to neutralize 1 gram:

Jones	0.30 to 0.66;	average, 0.51
Hortvet	0.38 to 0.66;	average, 0.52
Bureau of Chemistry	0.26 to 0.68;	average, 0.43

VIII. ALKALINITY OF INSOLUBLE ASH.

To the platinum dish containing the insoluble ash from determination VI add an excess of tenth-normal acid (usually 10 cc) and about 30 cc of water. Boil gently

until solution is complete. Cool and titrate with tenth-normal sodium hydroxid, using methyl orange as an indicator. Subtract the number of cubic centimeters of tenth-normal alkali used from the number of cubic centimeters of acid and the remainder will be the number of cubic centimeters of acid used to neutralize the insoluble ash. Divide this number by 5 and the result is the alkalinity of insoluble ash expressed in terms of the number of cubic centimeters of tenth-normal acid necessary to neutralize the ash of a 1-gram sample.

Pure maple sugar—cc of acid necessary to neutralize 1 gram:

Jones	0.55 to 1.72; average, 0.91
Hortvet	0.66 to 1.46; average, 1.06

Pure maple sirup—cc of acid necessary to neutralize 1 gram:

Jones	0.36 to 0.94; average, 0.54
Hortvet	0.32 to 0.79; average, 0.61
Bureau of Chemistry	0.31 to 0.92; average, 0.52

IX. RATIO OF INSOLUBLE ASH TO SOLUBLE ASH.

This ratio is found by dividing the second result (per cent soluble ash) obtained under determination VI by the first (per cent insoluble ash).

Pure maple sugar—ratio of insoluble to soluble ash:

Jones	0.5 to 2.2; average, 1.1
Hortvet	0.5 to 1.4; average, 0.9

Pure maple sirup—ratio of insoluble to soluble ash:

Jones	0.7 to 2.6; average, 1.7
Hortvet	0.7 to 2.5; average, 1.4
Bureau of Chemistry	0.6 to 3.2; average, 1.8

X. REACTION WITH IODIN.

This test need only be applied when glucose is suspected to be present. The procedure is that outlined by Beckmann:[a]

Prepare a solution of the suspected sirup with an equal quantity of water; if dark in color, place a quantity of powdered animal charcoal in the tube and shake; allow to stand for a little while, and then filter. The resulting solution should be nearly colorless. To this is added a few cubic centimeters of iodin solution (iodin in potassium iodid). If glucose is present, the solution turns red or violet, the depth and character of the color depending upon the quantity and nature of the glucose employed for adulteration. A blank test with a pure sirup, using the same quantity of iodin solution, should be made at the same time for the purpose of securing an accurate comparison of color.

XI. POLARIZATION AT 87°.

This test need not be made unless glucose is suspected from the results of previous determinations or when commercial dextrose is supposed to have been added. It is based on the fact that at 87° invert sugar is optically inactive. Hence any positive rotation observed at this temperature is due to the presence of some optically active bodies other than invert sugar. The polarizations should be made in a jacketed silver tube, and in case of the polarization of invert solutions the acid should be just neutralized before trying to polarize. A strong right polarization of the invert solution is an indication of glucose. For determining the approximate amount of glucose

[a] Zts. anal. Chem., 1896, *35:* 267.

present the following formula can be used: Divide the plus reading of the inverted solution at 87° by 163 and multiply by 100. From the polarization at 87° the quantity of levulose in the reducing sugars can be also determined.[a] The reducing sugar present should be nearly equal parts of levulose and dextrose. If a large predominance of dextrose is found and a high content of reducing sugars, with low ash and no iodin reaction, the addition of commercial dextrose is indicated.

XII. MALIC ACID VALUE.

This determination is made by a modification of the calcium chlorid method of Leach and Lythgoe[b] and is carried out in the following way:[c]

Weigh 6.7 grams of the sample into a 200 cc beaker and add water to make a volume of 20 cc. The solution is made very slightly alkaline with ammonium hydroxid. Add 1 cc of a 10 per cent solution of calcium chlorid and heat to boiling. Then add 60 cc of 95 per cent alcohol, cover the beaker with a watch glass, and heat for one-half hour on a water bath. Remove and let stand over night. Filter, through a 9 cm No. 589 S. & S. filter, by decantation. Transfer precipitate to the filter by washing with hot 75 per cent alcohol and continue the washing until the entire filtrate measures 100 cc. Dry and ignite. Add from 15 to 20 cc of tenth-normal hydrochloric acid to the ignited residue, dissolve by careful boiling, cool, and titrate the excess of acid with tenth-normal sodium hydroxid, using methyl orange as indicator. The malic acid value is one-tenth of the number of cubic centimeters of acid neutralized by the residue.

The results obtained do not absolutely express the malic acid present, but give a "value" that is useful in determining whether the product is adulterated. This procedure need only be applied as a confirmatory test of adulteration, as the lead number gives practically the same information.

Pure maple sugar—malic acid value:

Jones	0.65 to 0.84; average, 0.75
Hortvet	0.98 to 1.67; average, 1.29

Pure maple sirup—malic acid value:

Jones	0.41 to 0.72; average, 0.53
Hortvet	0.84 to 1.76; average, 1.07

XIII. PERCENTAGES OF CALCIUM AND POTASSIUM OXID AND SULPHUR TRIOXID.

The complete analysis of the ash of pure maple goods shows it to consist largely of carbonates of lime and potassium, with relatively small amounts of magnesium, sodium, and silica. Sulphates and phosphates are also present in small and varying quantities. The

[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. 110, p. 17.

[b] J. Amer. Chem. Soc., 1904, *26:* 380.

[c] J. Amer. Chem. Soc., 1904, *26:* 1536; Bureau of Chemistry, Bul. 105, p. 16; Bul. 107 Rev., p. 74.

determination of the former is of use at times in detecting adulteration, since sulphates are present in relatively large amounts in many commercial cane and beet sugars. The amounts of lime and potash also afford data useful in judging the purity of suspected samples. Methods for these individual determinations are given in Circular 23 of the Bureau of Chemistry, entitled "Methods for the Examination of Maple Products," page 6. Owing to the work involved, these determinations are made only in important cases when other tests do not furnish convincing data.

Compiled analyses of ash of maple products and adulterants (Jones).[a]

No. of sample.	100 parts of ash contain—			Ratio[b] of—		
	CaO.	K_2O.	SO_3.	CaO to K_2O × 100	CaO to SO_3 × 100	K_2O to SO_3 × 100
	Per cent.	*Per cent.*	*Per cent.*			
MAPLE SIRUP.						
1	18.03	31.97	2.30	177	12.7	7.2
2	20.00	30.00	1.91	150	9.6	6.4
3	23.98	38.98	1.06	163	4.4	2.7
4	19.81	35.90	0.68	181	3.4	1.9
5	20.76	36.22	1.58	174	7.6	4.5
6	21.86	35.48	1.74	162	8.9	4.9
MAPLE SUGAR.						
7	23.16	25.69	2.42	110	10.4	9.4
8	31.74	18.26	1.67	57	5.2	9.1
9	23.01	29.04	1.51	126	6.6	5.2
10	21.03	32.95	1.67	153	8.0	5.1
SUMMARY OF MAPLE PRODUCTS.						
Minimum	18.03	18.26	0.68	57	3.4	1.9
Maximum	31.74	38.98	2.42	181	12.7	9.4
Average	22.34	31.45	1.65	141	7.4	5.3
BROWN SUGAR.						
11 (Light)	4.17	39.58	4.86	949	117	12
12 (Dark)	11.32	30.72	17.78	272	157	58
13 (Dark)	21.62	55.40	5.95	257	27	18
14	15.63	40.62	4.58	260	29	11
ADULTERATED MAPLE SIRUP.						
15	2.35	32.52	4.56	1,384	194	14

[a] Eighteenth Ann. Rpt., Vermont Agr. Exper. Sta., 1905, p. 331.
[b] In the ratios given in this and other tables following the antecedent term has been made the divisor.

XIV.—TABULATED SUMMARY OF RESULTS.

The figures in the following table are based on the results on pure maple products published by Winton, Hortvet, and Jones, and on some sixty samples of pure maple sirup analyzed in the Sugar Laboratory of the Bureau of Chemistry in 1904 and 1905. The

average has been calculated from all of these with the exception of the figures for the percentage of water, polarization at 20° and 87°, and invert sugar, for which only a few determinations were given and no averages were determined.

Summary of compiled determinations on pure maple sugar products.

[Calculated on original substance.]

Determinations.	Maple sugar.			Maple sirup.		
	Minimum.	Maximum.	Average.	Minimum.	Maximum.	Average.
Water.....per cent..	3.05	11.0	.....	Not more than 32.00		
Direct polarization.....do....	72.6	87.4	.....	51.0	62.2	.....
Invert sugar.....do....	1.16	8.37	.....	0.34	9.17	.....
Lead number.....	1.83	2.48	2.23	1.19	2.03	1.49
Total ash.....per cent..	0.64	1.32	0.91	0.46	1.01	0.60
Soluble ash.....do....	0.33	0.67	0.46	0.21	0.63	0.38
Insoluble ash.....do....	0.20	0.87	0.46	0.14	0.56	0.23
Alkalinity of soluble ash.....	0.40	0.95	0.63	0.26	0.68	0.50
Alkalinity of insoluble ash.....	0.55	1.72	0.94	0.31	0.94	0.54
Ratio of insoluble to soluble ash..	0.5	2.2	1.00	0.6	3.2	1.7
Iodin reaction.....	.....	.....	None.	.....	.....	None.
Polarization at 87°:						
Before inversion (°V): Same as sucrose.						
After inversion (°V).....	−2.0	+2.0	.....	−2.0	+2.0	.....
Malic acid value.....	0.65	1.67	1.01	0.41	+1.76	0.78

In a great many cases the analyses did not include a determination of moisture, so that the figures could not be calculated to a dry basis, as should be done by the food chemist, only such analyses being perfectly comparable. The following figures were taken from the Vermont Experiment Station report in 1905 and so calculated:

Determination on pure maple products (Jones).

[Calculated to dry substance.]

Determinations.	Maple sugar.			Maple sirup.		
	Minimum.	Maximum.	Average.	Minimum.	Maximum.	Average.
Lead number[a]	1.94	2.51	2.28	1.76	2.69	2.28
Total ash.....per cent..	0.71	1.47	1.01	0.77	1.32	0.92
Soluble ash.....do....	0.40	0.70	0.53	0.45	0.72	0.58
Insoluble ash.....do....	0.22	0.97	0.48	0.25	0.78	0.34
Alkalinity of soluble ash.....	0.44	0.89	0.68	0.46	1.02	0.79
Alkalinity of insoluble ash.....	0.61	1.91	1.01	0.55	1.45	0.83
Ratio of insoluble to soluble ash...	0.5	2.2	1.1	0.7	2.6	1.7
Malic acid value.....	0.72	0.93	0.83	0.65	1.11	0.82

[a] Winton, loc. cit.

CANADIAN MAPLE PRODUCTS.

There is imported into this country some maple goods, mostly sugar, from Canada. Usually this sugar is hard, dark-colored, and strong in flavor. It can stand considerable dilution with sugar, and still have a fairly strong maple taste, and it is for that reason that it is sought by blenders of maple goods. This does not compose the total importation, but the greater portion is of this class of goods.

Jones finds the Canadian maple goods to conform closely to our figures of analysis. The average figures for six samples of Canadian maple sugar are as follows:

Total ash (per cent)	0.76
Soluble ash (per cent)	0.47
Insoluble ash (per cent)	0.29
Alkalinity of soluble ash (cc)	0.57
Alkalinity of insoluble ash (cc)	0.67
Ratio of insoluble ash to soluble ash	1.6

The laboratory of the inland revenue department of Ottawa, Canada,[a] has made numerous analyses of maple products. Comparative figures are obtainable for one determination only—namely, total ash. For 185 samples of maple sugar the average total ash was 0.89 per cent, calculated to dry substance on a basis of 10 per cent of water or 0.80 per cent of ash in the natural products. The minimum figure was 0.52 per cent and the maximum was 1.44 per cent. For 182 samples of pure maple sirup the average was 0.77 per cent, calculated to dry substance on a basis of 35 per cent of water or 0.50 per cent of ash in the natural product. The minimum figure was 0.52 per cent and the maximum 1.38 per cent. A genuine product is considered by the Canadian department to have not less than 0.50 per cent of total ash and a malic acid value of not less than 0.40, figured to a dry basis.

INTERPRETATION OF RESULTS IN DETECTING ADULTERATION.

In the case of most samples the determination of the lead number and the percentage of total ash will give a clue as to the purity. If the results of these two determinations agree with the average figures given, it is safe to say that the product is either pure or has been most skillfully manipulated. If the sample fails under either of the examinations mentioned, further tests as to solubility and alkalinity of the ash are necessary. The most common form of adulteration is the addition of granulated sugar or a sugar sirup. By this procedure the percentage of ash will be lowered and also the lead number, and consequently all other figures will be proportionately decreased. The addition of brown sugar is practiced, but the very dark grades are not used to any great extent, as they influence the flavor. Brown sugar may be high in ash and consequently not change the percentage of total ash of the maple product, which will, however, show a different solubility and alkalinity, and by these figures the adulteration can be recognized. Again, the lead number and also the malic acid value will be greatly reduced by the addition of brown sugar. The percentage of sulphur trioxid

[a] Bulletin 140, p. 141.

will be high and also the ratio of calcium oxid to potassium oxid. It is possible in adulterating to bring the ash content and its analysis of solubility and alkalinity up to standard, but it is difficult at the same time to raise the lead number and the malic acid value. In such samples the percentage of potassium oxid, calcium oxid, and sulphur trioxid will indicate the sophistication.

In determining the proportion of ingredients, such as the amount of maple sugar and added cane sugar in a compound so labeled, the average figures given in the preceding table must be used to gain an approximate result, based on the deviation from them. In case granulated sugar is added, it is comparatively easy to obtain satisfactory figures; but if brown sugar and "ash dope" have been used the task is more difficult, though from a comparison of all the figures the percentage composition can be roughly approximated. The following table gives the analyses of adulterants commonly used. The determinations were made in the same manner as for maple products.

Analyses of the usual adulterants of maple products.[a]

Description of sample.	Total ash.	Soluble ash.	Insoluble ash.	Alkalinity of soluble ash.	Alkalinity of insoluble ash.	Ratio of insoluble to soluble ash.	Malic acid value.
Brown sugar:	*Per ct.*	*Per cent.*	*Per cent.*	*cc.*	*cc.*		
Dark	4.33	2.74	1.59	0.76	2.34	1.7	.....
Light	1.44	1.30	0.14	0.48	0.26	9.3	.....
Medium	2.80	2.15	0.65	0.15	1.18	3.3	.....
Light	1.06	1.00	0.06	0.30	0.16	16.7	.....
Light	0.74	0.68	0.06	0.26	0.15	11.3	.....
Very light[b]	0.59	0.46	0.13	.....	.....	3.5	0.08
Medium[b]	1.65	1.23	0.42	.....	.....	2.9	0.10
Medium[b]	1.81	1.42	0.39	.....	.....	3.7	0.18
Dark[b]	0.85	0.64	0.21	.....	.....	3.1	0.18
Raw cane sugar	0.59	0.41	0.18	0.32	0.46	2.3	.....
Filtered sirup from the same	0.26	0.16	0.10	0.24	0.24	1.6	(c)
Raw cane sugar	0.46	0.23	0.23	0.36	0.52	1.0	.....
Filtered sirup from the same	0.26	0.16	0.10	0.24	0.22	1.6	(c)
Raw cane sugar	0.32	0.10	0.22	0.18	0.42	0.5	.....
Filtered sirup from the same	0.19	0.11	0.08	0.22	0.24	1.4	(c)
Beet sugar:							
White	0.33	0.31	0.02	0.40	0.02	15.5	.....
Light	0.86	0.78	0.08	0.38	0.28	9.8	0.08
Karo[d]	1.03	0.86	0.17	0.40	0.40	5.0	.....
Confectioners' glucose	0.57	0.45	0.12	0.24	0.18	3.8	.....

[a] Eighteenth Ann. Rpt., Vermont Agr. Exper. Sta., 1905, p. 319.
[b] Hortvet, J. Amer. Chem. Soc., 1904, *26*. 1542.
[c] Average, 0.35.
[d] 85 per cent glucose and 15 per cent cane sugar.

The figures for total ash given in the table vary considerably among themselves, owing to different manufacturing conditions, and are, as a rule, higher than the figures for the total ash of maple products. In the figures for percentage of insoluble ash the variation is small and the figures themselves are lower than for maple products. Therefore the ratio of insoluble to soluble ash is much higher than in maple products. Only a few figures for the malic acid value are

given, but these are decidedly low as compared with maple products. Were the lead number determined on these samples, the lowness of the figures would be very noticeable. Hence the value of the lead number and the solubility of the ash in determining adulteration can be seen.

Adulteration with commercial glucose is readily recognized by the high direct polarization and the strong plus reading of the inverted solution at 87°. A reading of 2° or 3° to the right at 87° C. on the inverted solution would hardly indicate glucose, as at 87° there is liable to be a destruction of levulose. Hence great care should be exercised in making readings at that temperature, and the solution should be brought quickly to 87° and a series of readings made without delay. Again, the sample may have fermented, and this would influence the results, possibly to the extent of causing a small plus reading. However, the greatest difference would be noted in the sucrose content and amount of reducing sugar present, and therefore it is necessary to use precautions against fermentation, as the analytical results from such a sample are of little value.

[Cir. 40]

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